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**REPORT ON VISITS TO A SELECTION OF RESEARCH AND OPERATING
ESTABLISHMENTS IN THE FIELD OF PETROLEUM PROCESSING IN
ENGLAND AND FRANCE - OCTOBER 24 TO NOVEMBER 7, 1960**

by

J. P. Mogan

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REPORT ON VISITS TO A SELECTION OF RESEARCH AND OPERATING
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INTRODUCTION

This report includes a summary of the facilities observed at each of the establishments visited. For the most part, however, the information obtained took the form of unrelated techniques and "tricks" in pilot plant design which are of present or expected use in similar projects in the Petroleum Process Engineering Section, and thus are presented largely in point form. The installations visited are as follows:

Humphries and Glasgow Ltd. - Constructors

The Institute of Petroleum - London Branch

British Petroleum Ltd. - Kent Refinery

British Petroleum Ltd. - Sunbury Research Station

The Gas Council - Midland Research Station

The Dorr-Oliver Corp. - Soudes Place Research Institute

Shell Berre Lte. - Petit Couronne Refinery

L'Institut Francais du Petrole

Esso Research Ltd.

^{1/} Scientific Officer, Fuels and Mining Practice Division, Mines
Branch of the Department of Mines and Technical Surveys, Ottawa.

I VISITS TO BRITISH INSTALLATIONS

Visit to Humphries and Glasgow - Constructors, Victoria Station House, London

Mr. N. H. Williams

Humphries and Glasgow have recently completed the construction of their first pilot plant for the "Midlands Gas Council". This project, for oil gasification in a fluidized coke bed, was undertaken at cost as a result of successful smaller scale studies by the Gas Council in the expectation of future commercial utilization. Recent reductions in the benzene and sulphur markets have since rendered the process of less immediate commercial interest.

This process, involving the fluidized gasification of oil in a circulating coke bed is carried out at up to 1000 psi and 900°C in a 2 ft 6 in. diameter inconel reactor. The entire inner reaction shell is contained in a cold pressure vessel separated from the hot shell by loose "alundum" fill. The coke provides both reaction sites and heat transfer uniformity. The recirculating gas is conditioned by hot potassium carbonate and lead acetate scrubbing for H_2S removal. The efficiency of this treatment was responsible for an interesting difficulty in the initial operation. The unexpected elevation of the furnace temperatures was traced to the conversion of the residual carbon monoxide to carbon dioxide. The solution took the form of re-adding a small amount of hydrogen sulphide to poison the nickel alloy furnace tubes which were exerting a catalytic effect.

Standard groove and spigot mechanical fittings were used throughout the equipment; it was found necessary to heat and cool very slowly to avoid leakage due to thermal gradients. In addition, insulation was

required at some distance from the heated zones to minimize the extreme thermal diffusion effects of high pressure hydrogen.

Visit to Esso Research Limited, Abingdon, Berkshire

Dr. P. J. Agius
Dr. A. Winward
Mr. G. Woodward

The writer was first given a brief tour of the analytical facilities at Abingdon - these consist of: spectrograph, mass spectrometer, gas calorimeter, microanalysis laboratory, and conventional petroleum testing facility. A brief tour of the engine testing operations followed in which a variety of engines are load tested for lubricant evaluation, fuel effect on valve performance, etc. Various automobiles are being road and dynamometer tested on the same basis. A six cylinder railway diesel is operated for fuel and lubricant testing; the main problem accruing from this project was the absorption of the power produced.

There followed a detailed tour of the pilot plant laboratories, which comprise four batch stills, two continuous vacuum stills, three hydrodesulphurization units of various pressure ratings, five batch reactors, a steam cracker, dewaxer, stripper, copper sweetener, clay treater, and a stacked stage extractor.

During this tour a number of points were noted:

1. The stills were induction heated in some applications as this method restricts the heat to the area desired. Initially, the performance of the installation was below expectations, but this was corrected by cooling the induction coils.

2. Heater control was generally by off-on instruments of the "PyroVane" type. Difficult applications warranted West Ll40 controllers

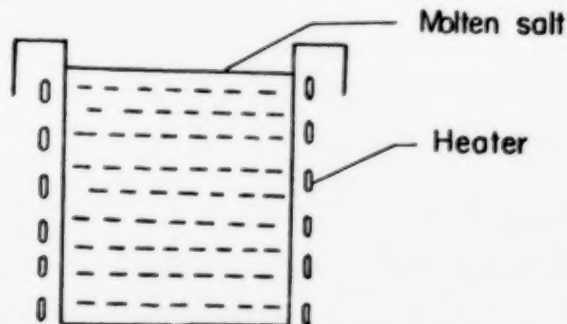
which provide a non on-off type proportional control.

3. A variety of preheater methods were employed on the continuous plants:

The high pressure hydrodesulphurizer (1600 psi) used aluminum blocks cast around tubing coils and strip heaters. These yield adequate heat exchange but are difficult to replace in the event of burned out heaters.

For the intermediate hydrotreater (800 psi) a 41 ft coil and resistance heaters were immersed in an eight inch diameter fluidized sand bed through which 3 - 4 cfm of heated air was passed. Temperature control was through a central well with "Pyrotenax" couples; the well has since been discarded to improve response.

A standard salt pot was the method generally employed on the other equipment, and has proved the most trouble free after the addition of a lip to discourage salt creep to the heaters.



4. Gas to the high pressure hydrodesulphurization unit was compressed by a "Dr. Thedie of Berlin" high pressure pump to 1600 psig. Gas supply was at 1 - 2 psig from cylinders and was measured on a pressure volume relationship. A platinum catalyst installation was in progress on the suction system to perform the dual function of a warning device (from the thermocouple detected temperature rise) and an oxygen

removal device until corrective action could be taken. It was mentioned that the above gas pump had been known to seize if run in on nitrogen.

5. The product separator levels (high pressure hydrodesulphurization units) were controlled with differential pressure cells and Hammel Dahl Size 15 trim control valves. The valves were arranged to open on signal failure for safety reasons. Lab samples were withdrawn slowly from the high pressure separators by means of preset needle valves and block valves. The high pressure product was de-gasified by spraying it into low pressure receivers, the levels of which were visually controlled with needle valves.

6. The high pressure hydrodesulphurization reactor was a one inch inside diameter cylinder with a central thermocouple well packed with 1/16 inch Peter Spence cobalt molybdate extrudate. Hydrogen passed through the reactor once and was vented.

7. The low pressure hydrodesulphurizer (350 psig) recycle gas stream was scrubbed with 8 percent caustic solution supplied from a pump-around system with a spring loaded by-pass valve. The scrubber levels were maintained with conventional differential pressure cell - control valve systems.

8. Oil feed was generally by means of "Bosch" type variable fuel injection pumps with gas cushions to dampen fluctuations.

9. Graphite rupture disks were found to be reliable low pressure safety devices.

10. Air operated drum pumps were said to perform well with high viscosity materials.

11. 18-8-Mo stainless steel was employed even in non-corrosive applications for ease of cleaning and storage.

12. Hermeto fittings were generally used.
13. All equipment operating above 1500 psi was shielded.
14. Tongue and groove seals with compressible asbestos gaskets were generally used; 2 degree interference cones were employed in difficult applications.
15. To ensure reliable gas temperatures, 1/16 inch "Pyrotenax" thermocouples six inches in length were employed to minimize conduction errors. These extended vertically downward into piping sections to eliminate sagging. It was found that iron-constant thermocouples of the "Pyrotenax" type yielded predictable calibrations but chromel-alumel did not.
16. The high pressure vent gas was controlled in two stages - an automatic pressure regulator followed by a needle valve. A 3/4 inch diameter vertical pipe before the regulator was found to eliminate oil mist problems.
17. Work was being carried on to adapt the batch fractionator to continuous co-current operation with the high pressure hydrodesulfurization unit.
18. A program to study the effect of asphalt spray on moisture conservation in desert areas was in progress.

Monthly Meeting of the Institute of Petroleum, London Branch

Mr. A. W. Deller of the public relations department of Shell Oil Limited spoke on his profession as applied to the oil industry. He outlined the difficulties of this international industry in presenting a unanimous viewpoint in view of conflicting geographical interests, and also the undesirability of such because of the danger of creating the impression of a cartel.

The speaker dealt with the necessity of selling public relations to management, and lamented the lack of an oil company "image" in the general public's mind. He then discussed some of the steps that should be and were being taken to build this "image".

Visit to British Petroleum Limited, Kent Refinery, Isle
of Grain, Kent.

Mr. A. R. Stark - London Office
Mr. C. P. Williamson - Technical Chief, Kent Refinery
Mr. Bacon - Kent Refinery

The British Petroleum Refinery, located on the estuary of the Medway River has a crude oil capacity of $4\frac{1}{2}$ million tons per year. Deep water jetties are provided for crude unloading, as well as docking facilities for water product transport. The salt water cooling system comprises an extensive concrete moat and a reservoir created by isolating a small bay. Effluent water is divided into normal and heavily contaminated streams, and is thoroughly conditioned before returning to the Medway.

Two atmospheric distillation units are operated embodying primary and secondary columns, and are capable of processing $4\frac{1}{2}$ million tons per year. In conjunction with these, two vacuum units with a capacity of one million tons per year provide lube and cat cracker stocks. The catalytic cracker is a side by side unit using a powdered silica-alumina catalyst. Two catalytic reformers, with platinum catalyst, and a thermal reformer upgrade distillate to aviation gasoline and motor gasoline respectively while a catalytic alkylation plant (sulphuric acid catalyst), and an isopentane fractionator produce high octane motor gasoline.

Kerosene is de-aromatized in a liquified sulphur dioxide extraction plant. Sulphur is removed from distillation and reformer feed in

a fixed bed hydrofiner, from which the hydrogen sulphide produced is removed in an amine scrubber and converted to elemental sulphur over a bauxite bed. The lubricant stocks are propane de-asphalted, furfural extracted, and methyl ethyl ketone de-waxed. A complete distribution center is operated at the refinery site.

The specific points of interest noted in the refinery are as follows:

1. The fluid catalytic cracker, with side by side reactor and regenerator, utilizes one spent catalyst downcomes and air lift to regenerator, and two fresh catalyst lines from the regenerator to the reactor. Oil and dispersion steam feed is to the bottom of the fresh catalyst lift ducts just past the slide valves; because an estimated 75 percent of the conversion occurs within these ducts, steam demand has been reduced from the design value of 2 x 900 to 2 x 90 lbs per hour.

The reactor operates at 10 psig and 930°F, the regenerator at 5 psig and 1100°F; both are equipped with pressure taps across the bed to indicate bed level, and taps in the bed for bed density. The reactor bed level is controlled through the spent catalyst slide valve which is actuated by the differential pressure across the reactor bed. The slide valves in the fresh catalyst lines however are controlled by the reactor temperature.

Catalyst attrition rate is about 0.15 lbs per barrel of feed, and balances make-up requirements from iron contamination. The product is fed to a fractionator equipped with baffle plates below the feed tray, and bubble caps above. The fractionator bottoms with entrained catalyst are returned to the reactor. Pressure taps on the unit are of the double side inlet form, with the end of the tap containing a plug for rodding out. Taylor, Short and Mason miniature recorders are employed for pressure and gas bulb temperature measurements.

2. The hydrofiner, employing two groups of three reactors in series, has a capacity of 10,000 barrels per day of 2.5 percent sulphur gas oil. The reactors contain Peter Spence cobalt molybdate extrudate and operate at a space velocity of 2.5 at 750°F and 300 psig. The pressure is fixed by that of the hydrogen stream available from the cat.reformer; the activity of the catalyst was found to change little in the range from 250 to 700 psig. Regeneration is carried on with steam and air at 800°F at atmospheric pressure and occurs with variable frequency dependent on the feed processed.

3. Gas samples are taken throughout the refinery in soccer ball bladders (adequate except for samples containing significant amounts of butene) and sampled from the bladders by hypodermic syringe.

4. Plumbing ball valve washers are employed as self sealing diaphragms through which gas samples are introduced.

5. Gas chromatography columns are fabricated from nylon tubing and employ a pilot hydrogen flame in an oxygen excess. Gas constituents are then detected by temperature peaks indicated by a thermocouple above the pilot flame.

6. An automatic spectrograph is employed to detect barium and calcium percentage to determine additive concentrations in lube oils.

Visit to British Petroleum Limited,
Sunbury Research Center, Sunbury, Kent

Mr. P. Fleming - Research Chief
Mr. W.N.N. Knight - Assistant to Research Chief
Mr. E. H. Porter - Technical Development Chief
Mr. R. O. Jodd - Chief, Mechanical Design Section

As the information derived from this visit is of wide scope, and in most cases constitutes isolated points which promise to be of use in pilot plant construction, the summary will be presented simply as a list.

1. The use of bauxite beds before fixed bed hydrofining installations to remove heavy metals and mineral matter was discussed, and the patent numbers covering this field forwarded.

British Patent No.	729,301
" " "	729,303
" " "	735,520
" " "	735,521
" " "	737,572
" " "	744,045
" " "	767,122
" " "	786,435

2. The spray introduction of heavy feeds to fluid beds was suggested.

3. York mist eliminators are operated at 5 ft per sec by B.P. with marked success.

4. The research carried on at Sunbury has prompted a suspicion that volatile (excess of 600°C) vanadium compounds exist.

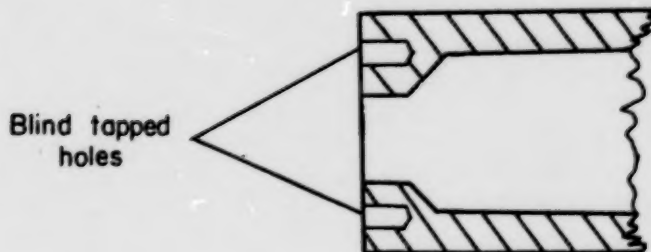
5. High sulphur coke de-sulphurizes spontaneously in an inert atmosphere at 1500-1600°C.

6. Based on experience at Sunbury with "Gama Ray" bed level detectors for fluid units, our application is judged marginal because of thick reactor walls.

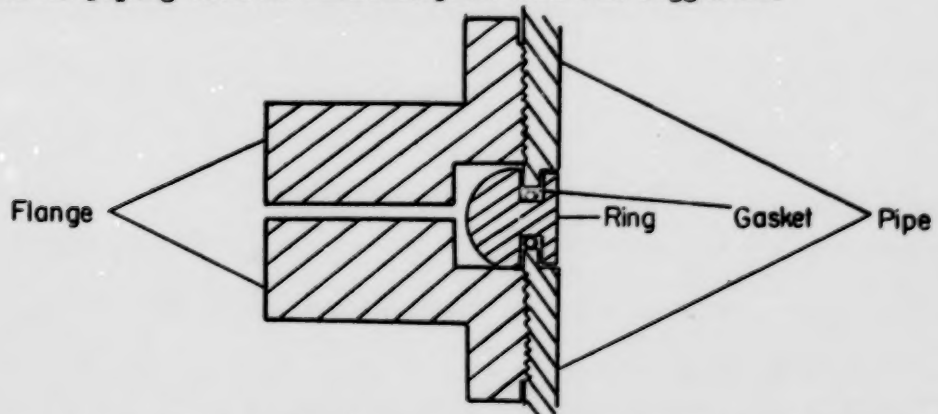
7. Off-on type Bailey temperature controllers in conjunction with variacs provide accurate temperature control for the pilot fluid catalytic cracker.

8. The most satisfactory seal employed is of the tongue and groove type with crushable jacketed asbestos gaskets. All bolts are specified superstrength on the basis that a torque limit would eventually be violated in an effort to effect a seal. Crane "Thread Guard" is found to effectively prevent thread galling. Ermeto fittings have been used up to 1200°C at Sunbury. The use of gas filled metal "O" rings has been unsuccessful.

9. Leak free operation of heated high pressure piping systems frequently depends on the provision of expansion devices.
10. A large monoblock vessel should be necked down to provide a four inch (hand size) top opening to eliminate the need for large flanges.



11. The use of a seal ring with crushable gaskets to join two pieces of piping with an uninterrupted wall was suggested.

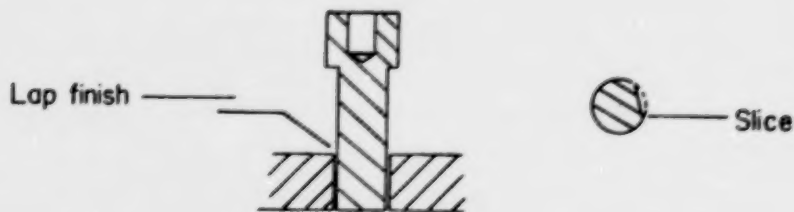


12. The high thermal conductivity, and consequent heat loss of hydrogen streams in pilot plant applications has resulted in the policy at Sunbury of supplying unheated gas to reactors where possible, as the small heat capacity of the gas relative to the oil feed often produces minimal cooling. Alternatively feed and gas streams are mixed before heating, and if it is necessary to heat gas alone, the piping runs are kept as short as possible.

The use of a heat sink type of heater, such as a lead bath, was recommended to minimize ambient effects. In general, the values

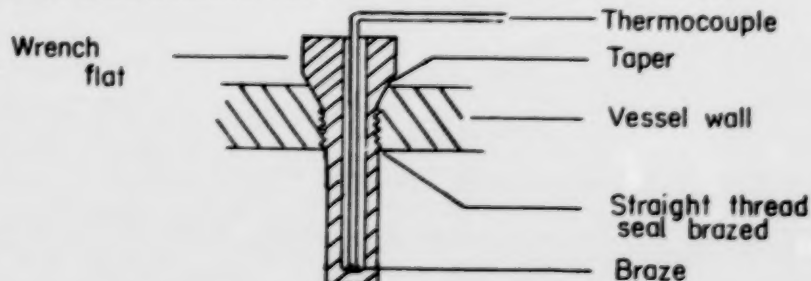
K = 1 for all gases and K = 30 for all oils are employed at Sunbury for safe pilot plant design of heaters. When employing a lead bath, the fact that the coil and heaters will be bouyant in molten lead should be considered, graphite should be sprinkled on the surface to retard oxidation, and the bath should be de-drossed about once per three months.

13. If a stock miniature control valve is not available for a particular application, custom trim is fabricated at Sunbury by lap finish- ing a plug and port and removing thin slices from the side of the plug until the desired characteristic is achieved.

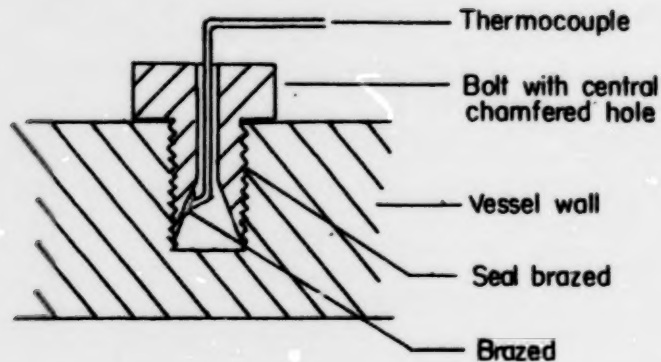


Extremely fine control may be obtained by cutting a disappearing thread on a lap finish plug and seat combination. The "Anrin Wee Willie Domotor" control valve is endorsed by the Sunbury design section. The use of stainless steel throughout the pilot plant systems minimizes the risk of clogging miniature final control elements with scale.

14. Thermocouple installations at Sunbury employ 1/16 inch diameter "Pyrotenax" type couples supplied by the West Instrument Company. For internal and skin measurements they are installed as shown below.



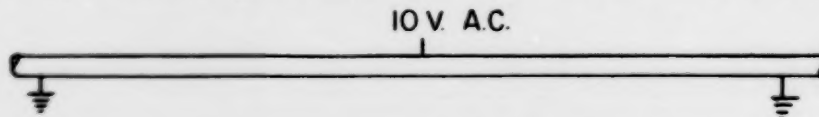
Internal Temperature



Skin Temperature

No difficulty has been experienced in operating the above methods at temperatures in excess of the softening point of the brazing material, as the semi-solid braze still resists being squeezed out of the thread. The temperature measurement provided by these systems is adequate for pilot plant purposes, since in general they have found that refinery temperatures are only reliable to $\pm 12^\circ\text{F}$.

15. A convenient type of heater employs the pipe itself as the resistance element to which a low voltage high current source is provided from a transformer. The power tap must be located in the center of the line however, otherwise grounded pumps and valves would be heated.

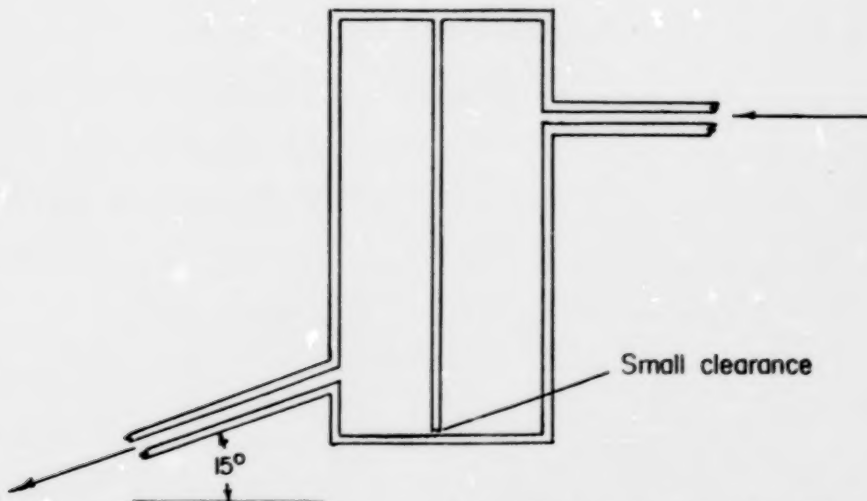


16. For pilot plant heating applications, it is usually found impractical to design for turbulent flow. Electrical heat flux however is kept to 10 watts per square inch, or 20 wpsi in emergencies. A flux of 40 wpsi invariably leads to difficulties.

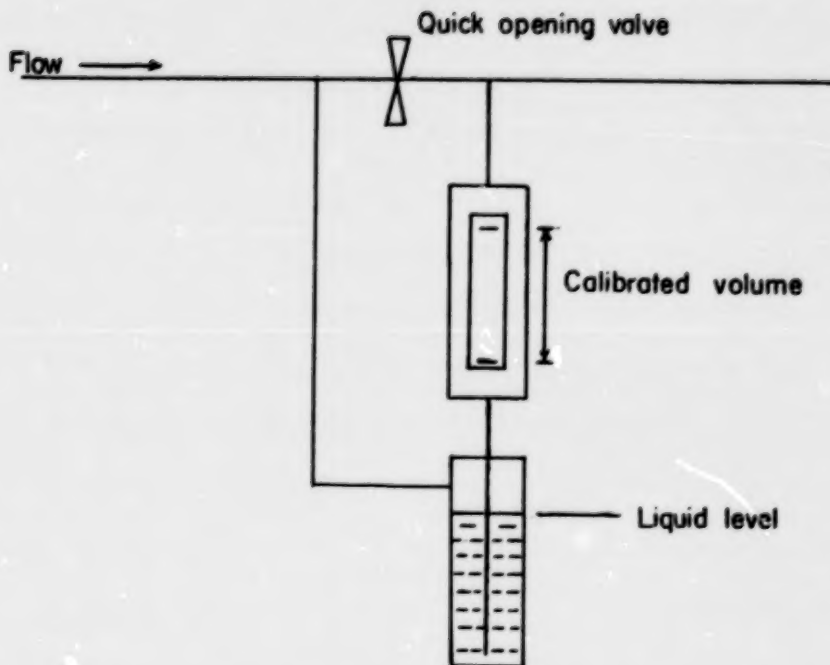
17. A flow measuring kit (supplied by Foxboro) is in use in which the body of a differential pressure cell contains a flow restriction on one side of the diaphragm across which the gas stream flows, thus

combining orifice holder, connecting lines, and signal transmitter in one case. It has been found that the orifice is the most reliable flow measuring device; extremely small flows may be measured using a jewel watch bearing peened into a brass plate as an orifice.

18. The "Corblin" diaphragm pump is favoured as an oil feed pump, gas compressor, and gas recirculator. Difficult gas pumping problems can be solved by locating the pump valves remote from the pump and utilizing the oil feed as a valve lubricant. For small flows, a "Bosch" type variable fuel injection pump is used to hydraulically actuate a "Corblin" diaphragm pump. Direct current motors are generally preferred as a variable speed pump drive. The pulsation damper shown below effectively reduces the pulsations in a gas or liquid stream from a reciprocating pump.



Accurate instantaneous gas flow measurement may be obtained with the dual chamber device shown below.



The upper chamber is sized so that it will fill in 10 to 20 seconds, and is large enough to contain all the liquid in the lower chamber.

19. It was stated that derivative control had never been found applicable to pilot plant installations because of the proximity of instrument to control element and the size effect on lag. Several examples in which the use of derivative control had produced continuous cycling were pointed out.

20. The pilot fluid catalytic cracking unit employed side by side construction with a 4 inch I.D. reactor and 8 inch I.D. regenerator. The pressure taps, angled at 30° to the vertical to minimize clogging, conventionally indicate bed levels by the differential pressure across the vessels, and bed densities by differential pressure across the lower segment. The catalyst rate to the regenerator is controlled by the differential pressure across the transfer line.

The slide valves, of similar design to those of Gulf Research and Development, employ purged guide slots in the gate to minimize clogging, with a clearance of 20 thousandths of an inch between the gate and sleeves.

It was recommended that, as general practice, fluidized solids control equipment should be designed so that the minimum opening is greater than five times the largest particle diameter to avoid bridging.

The 10 micron ceramic filters, located in the top of the reactor after the re-entrant stripping stream, are arranged on an automatic purging cycle of 15 minutes purge and 45 minutes operation for each of the four filters.

Visit to the Midlands Research Station, The Gas Council,
Birmingham Research Station, Solihull, Warwickshire.

Dr. F. J. Dent
Dr. L. A. Moignard

The Midlands Research Station operates a fluidized coke bed hydrogasifier in which light, middle, and heavy oils are cracked to produce a product gas stream of 500-800 BTU/cu ft. The experimental apparatus is designed for operation at pressures up to 70 atmospheres and temperatures to 900°C, and employs a 90 micron fluidized coke bed as a heat exchange medium. Preheated (500-550°C) hydrogen, obtained by steam cracking a portion of the product gas, is reacted with the oil feed in the fluidized bed, and the resultant gas cooled in a heat exchange and recycle quench system, followed by steam iron-oxide CO₂ removal and Benfield H₂S absorption.

Most of the work has been applied to light and middle vapourizable oils at 700-800°C and 25 atmospheres. The experiments involving heavy distillates and crudes have demanded refined feeding techniques;

the oil is atomized into a disperse phase moving region of the coke bed to prevent agglomeration and caking. (Superficial gas velocities of about 20 fps are utilized in the feed region, as compared to about 0.5 fps in the main bed). A coke laydown of about fifty percent of the Conradson carbon value of the feed oil has been observed. This requires a lock hopper arrangement for periodic removal of the excess coke.

If operated at oil to gas ratios of 3 - 5 gallons per 1000 cu ft, the aliphatic constituents of the feed stocks tend to gasify entirely, while the aromatics are stripped of their side chains, leaving a condensate rich in pure simple aromatics. A feed ratio of 20 gallons per 1000 cu ft results in a conversion of 15-20% of the aliphatic constituents to monocyclic aromatics, providing a means of adjusting the operation to benzene demand. The process has been found to be self sustaining for light and middle oils, and to require supplementary heat for the processing of heavy distillates and crude.

The following are points of interest observed in the construction of the plant.

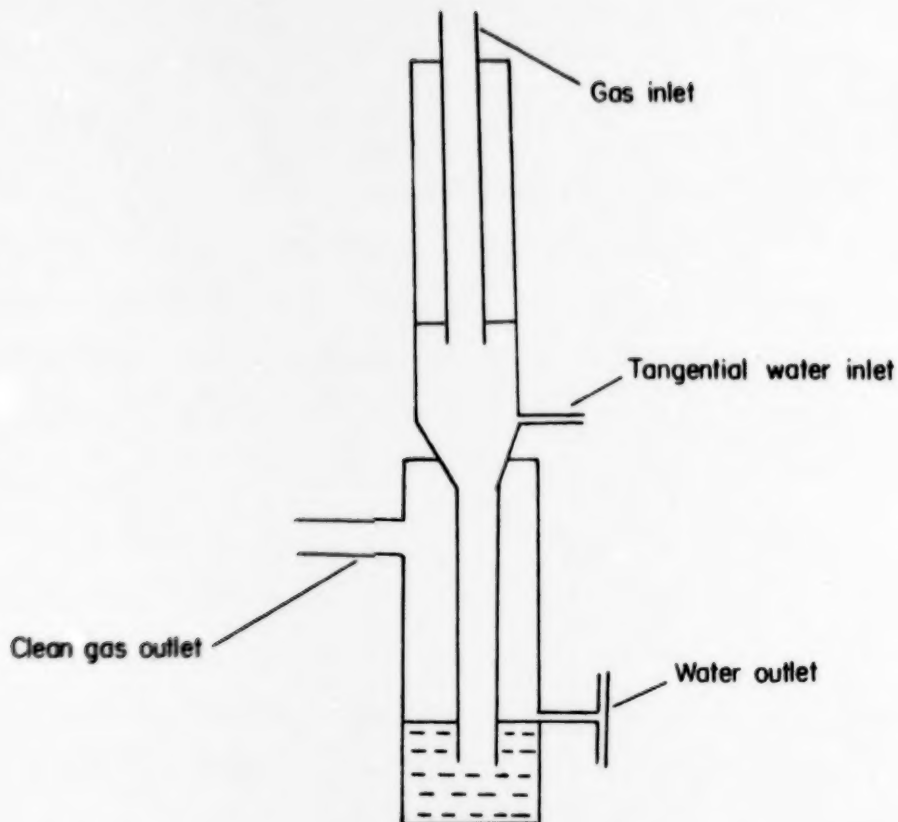
1. The internal plant of all welded construction is surrounded by a non-heated pressure retaining shell, all connections being through dual flanged pipes. The wide thermocouple and pressure taps originally led through bellows arrangements to allow for expansion, but this has since been discarded due to leakage. The present system employs a three element sandwich top flange with the previously mentioned dual insulated connections to the process piping and central thermocouple well.
2. Sealing on the mild steel pressure shell, which is kept below 200°C through the use of loose "Alundum" insulating fill, is by means of tongue and groove flanges with crushable metal clad asbestos gaskets. The flange bolting is checked for length after each run to prevent leakage.

3. The quench cooler embodies a circulating condensate stream in a "Rashig Ring" packed tower with a water cooler removing heat from the quench liquor. Secondary gas stream cooling is accomplished in a heat exchanger of novel design; the cooling water overflows through a vertical shell with an open top, thus eliminating the need for a floating header, and providing visual evidence of water failure by steaming.
4. The use of superstrength "Nemomic D.S." alloy has been unsuccessful due to unexpected sulphur corrosion. Stainless steel heater tubes of 25-20 alloy have exhibited welding difficulties.
5. A two stage centrifugal pump in a pressure casing provides quench liquid circulation, while an atmospheric fan in an external pressure case moves the high pressure hydrogen.
6. In critical locations, the use of plug valves is recommended because of the eroding effect of dusty high pressure gas streams on other types of valve seating if a perfect seal is not obtained.
7. A plexiglass model of the internal vessel was constructed to study catalyst flow patterns.

Visit to the Soudes Place Research Institute,
Dorr-Oliver Corporation, Dorking, Surrey.

Mr. D. W. Beeken
Mr. Dennis

The Dorr-Oliver Company operates a versatile fluidized bed reactor and dryer combination which are used for pilot plant studies of immediate industrial application. Both units are heated by combustion gases from a ring burner rising in the annular space between the vessel and the lagging (dryer) or brickwork (reactor). Oil feed to the equipment is by "Bosch" type fuel injection pumps; control is by calibrated multiple orifices arranged so that a clogged orifice may be detected visually by a sight glass on the by-pass line of a spring loaded valve. Mr. Beeken stated that their experience had been that heavier oils were more readily introduced to fluidized beds than light oils, because of their tendency to coat the particles. Two to three cfm of gas per lb. of oil are employed by Dorr-Oliver for oil atomization. The effluent gas from the fluidized beds, after passing through settling and condensing chambers, is brought through a wet cyclone of the design shown below. This resulted in a reduction in caking problems and a very clean off-gas.



The study being carried on at present at the Soudes Place Research Institute concerns feeding problems involved in drying a wet solid with an angle of repose of greater than 90° . The approach taken is to provide a sloped hopper with the bottom fabricated from a steel diaphragm equipped with an air vibrator. A pulsating air current (provided by a motor driven stopcock) is employed to inhibit bridging.

An interesting pilot plant technique was mentioned in that non-standard rotameters could be fabricated from straight glass tubes containing central tapered rods and doughnut shaped floats. On occasion, the research institute has fabricated simple but effective dipleg cyclones by bending the top of a piece of straight tubing to form a tangential inlet.

II VISITS TO FRENCH INSTALLATIONS

Visit to the Refinery of Shell Berre at Petit Couronne
Rouen, France.

Mr. Richard - Technical Director
Mr. LaRocher - Assistant Technical Director

The refinery at Petit Couronne is a versatile complex, processing Venezuelan, Middle East, and Saharan crude. Great precautions are taken with the Venezuelan crude to avoid processing conditions which would have an adverse effect on colour as it provides the lube oil stocks.

The crude is distilled in three units: the old atmospheric section at 2400 tons per day, the new atmospheric section of 3600 ton capacity featuring internal neutralization, and a vacuum section of 2900 tons per day. The new atmospheric section employs a pump around system on two drawoff trays for neutralization to prevent caustic from reaching the lower section of the unit. This minimizes asphalt foaming, and restricts caustic usage to fractions to which it is beneficial.

A lubricating oil plant for treating the straight run vacuum lube stocks from the Venezuelan crude employs a furfural extraction plant, and acid and clay treaters. Hard bitumens are produced by vacuum redistillation; a cut back and blending plant, as well as asphalt blowing facilities are operated.

The catalytic reforming plant is composed of a platformer and unifiner plus fractionator. A catalytic hydrodesulphurizer utilizes platformer excess hydrogen and provides platformer feed. A thermal reformer and a fluid catalytic cracker plus fractionator treat feedstocks. Hydrogen sulphide is removed in a standard "Girbitol" plant, a "Doctor" plant treats gasoline, and a kerosene acid washery is operated. A feature of the refinery is the completely automatic tank farm, in which

all the valves of the manifold system were actuated electrically, and outage measurements of all tanks were indicated on a central consol by a capacitance device.

The catalytic cracker is a standard side by side unit, employing a regenerator vent gas reboiler as stripping steam supply. The regenerator temperature is controlled by torch oil injection, while reactor temperature is modified by steam injection and by varying the proportion of hot to cold feed. The total pressure on the unit is controlled at the fractionator off-gas stream and the regenerator vent gas stream, with the reactor pressure balanced between these two. The stripper contains an emergent gas cyclone, with the stripping gas being re-introduced to the main stream before the reactor cyclone.

The platformer is capable of reforming 1700 tons per day of naptha feed with a gas make of 14 percent. The platinum catalyst on a silica-alumina support has a one year life, and is recoverable to approximately sixty percent of its value. Platformer feed is treated in the hydrodesulphurizer at 60 atmospheres and 390°C employing a cobalt molybdate catalyst manufactured by Pechiney. This HA-102 catalyst is in the form of 3 mm extrudate and exhibits greatly enhanced activity over HA-101 which is supplied as irregular spheres. This increase is attributed to the physical properties of the new shape rather than composition modification.

An asphalt blowing pilot plant is operated at the refinery to evaluate optimum temperatures and blowing times for various asphalt stocks. Asphalt is pumped from a heated feed drum through a low voltage integral pipe preheater (200 amps at 20 volts) to the bottom of a four inch resistance heated tower. The feed rate is measured by outage on a small feed cylinder and is set by a tachometer equipped variable drive

operating a small centrifugal pump. Heated air is introduced at the tower bottom and escapes through coolers to an emulsion separator before venting. The blown asphalt overflows to a cooler and sample receiver.

Metallurgically, the refinery is constituted as follows:

Catalytic cracker:	Reactor body AISI 304 clad AISI 405
	Reactor grid APS 10 M4 (Pompey)
	Reactor grid pot (anti erosion device)
	PM-35 (Pompey)
	Reactor standpipes and risers MSVL (Pompey)
	Reactor cyclones ASTM 204 (refractory lined)
Unifiner:	Reactor ASTM 264 gr 5 clad ASTM 204
	Piping ASTM 199 TS
Hydrotreater:	Reactor AISI 321 clad AISI 204
	Heater ASTM 213 T 321

The valve position indicators employed at Petit Couronne were of interest for graphic panel use. Supplied by Evershed Vignoles Ltd., they consisted of moving bars behind frosted glass panels.

Visit to L'Institut Francais Du Petrole, Reuil-Malmaison, France

M. Thonon - Chief of Division of Applied Chemistry and Refining,
 M. Lavergne - Pilot Plant Design
 M. Verwaerde - Heart Pulse Processing

As the institute operates extremely varied and versatile pilot plant and laboratory facilities, no overall survey was attempted. Rather, those facets of the installation which were of immediate interest were studied.

The first of these was the high pressure hydrodesulphurization unit which operates at 1000 psi and 350 to 450°C. This unit embodies a co-current fixed catalyst bed through which oil is circulated at about 8 litres per hour. Normal recirculating hydrogen rates are maintained with make-up gas supplied from two 50 litre cylinders at 160 atmospheres.

Oil is pumped from a heated reservoir to the unit by a "Bosch" type fuel injection pump with a manually set feed rate. The rate is measured in a jacketed expanded glass section of the inlet line with calibration marks etched on the restrictions at either end. The hydrogen recirculation rate is determined with a 0.3 mm beveled orifice and manometer combination. (The manometers, Jergusen high pressure sight glasses, are protected by wire mesh and $\frac{3}{4}$ inch plexiglass panels).

Gas and oil are mixed before the preheater - a lead bath with oil coil and resistance heaters. It was stated that a high differential temperature is required between the bath and the gas-oil outlet because of the difficulty in heating high pressure gas streams. The reactor, of type 316 stainless steel, employs a central 8 mm thermocouple well and recessed crushable gasket end seals (tongue and groove construction is preferred) and contains cobalt molybdate catalyst. The hot products and hydrogen leave the reactor through a type 321 stainless steel heat exchanger at 300°C which is exhibiting rapid unexpected corrosion. Clogging in the lines from the exchanger due to the presence of a compound believed to be $S(NH_2)$ was eliminated by the addition of hot water jackets.

The recirculating gas and liquid product pass into the dual separator system, the first part being a simple pot, the second employing a side inlet and a top outlet equipped with a ceramic or sintered metal filter. Levels in both separators are detected with Jergusen sight glasses and controlled manually by means of needle valves.

Oil free gas from the separators is piped to the hydrogen sulphide absorption system. This consists of a 48 inch by 2 inch packed tower through which light gas oil is circulated. The gas oil, having absorbed the H_2S by virtue of its high solubility under pressure, is then passed through a reducing valve to an atmospheric

reservoir for recirculation. The gas that collects over the oil is vented through a caustic bottle. The H_2S free high pressure hydrogen is recycled by a "Corblin" diaphragm pump. (The life of the compound diaphragm in this service has been 50 to 5000 hours with an average of 2000 - 3000 hours).

A low pressure hydrodesulphurization unit is also operated at the institute. This unit is similar to the high pressure equipment, but is in part constructed from industrial glass piping. The preheater is of different design, employing a 2 mm I.D. coil immersed in a lead bath in a brass pot which had been cast around a resistance heater. Glass was not recommended for a pilot plant of this type because of breakage difficulties.

Experiments with a large fixed bed hydrogenator -5 inch diameter by 36 ft length - uncovered problems resulting from the occurrence of void spaces within the column on random packing. In time, these voids would tend to agglomerate, and high catalyst attrition rates would occur due to individual pellets swirling around in them. Accordingly, the top of the bed was packed with alloy steel balls to provide a floating load which tended to eliminate the voids.

A moving bed hydrogenator was constructed for atmospheric pressure operation. The rate of carbon laydown under the conditions employed required constant regeneration, so the catalyst pellets were dropped through the reaction zone and conveyed up through the regeneration zone by a mechanical elevator.

An investigation to determine the effect of the application of the "Pulse" reaction principle to liquid phase hydrogenation has been completed at the institute. This system employs a plastic and fabric bellows about six inches in diameter actuated by an eccentric

rod fixed at a rotating wheel. Adjustment of the rotational speed and eccentricity produces varied pulsing. For the hydrodesulphurization experiments it was necessary to provide a two foot section of oil filled tube with a cooling jacket to protect the bellows, and enclose the catalyst in five inch high cylindrical chambers fixed firmly in the reactor to prevent the entire bed from moving up and down the column.

The experiments were carried on at constant oil-gas rates, temperature, and pressure with pulse frequency and volume varied. This yielded the result that 200 - 2 inch strokes per minute achieved optimum efficiency, which was such that 1% sulphur feed stock was desulphurized to 0.15% in a static bed and 0.085% with pulsation. Unfortunately, the catalyst attrition rate was very high, amounting to 12% of the bed in 200 hours. As improvement in reaction efficiency was not balanced by the necessary energy input plus catalyst loss, the investigation has been discontinued. The principle, however, has proven effective for gas-liquid and liquid-liquid contacting, and research in this field is continuing.

CONCLUSION

The personnel of the installations visited in England and France were found to be exceedingly co-operative in describing their pilot plant techniques, experiences, etc. Many of these were of general applicability, while others were derived from equipment in which the problems encountered were similar to those encountered in installations proposed, in construction, and in operation at the Fuels Division. A variety of ingenious "tricks", and also information concerning unsuccessful techniques which have been tried and discarded, which cannot be found in the publications of the establishments concerned were learned during these visits.

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